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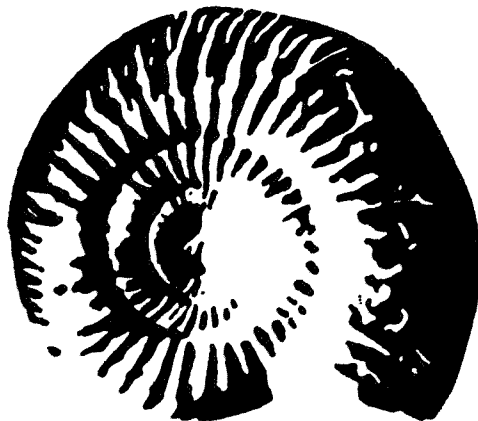
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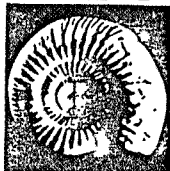
IKU



INSTITUTT FOR
KONTINENTALSOKKELUNDERSØKELSER

Continental Shelf Institute

Håkon Magnussons gt. 1 B
Postboks 1883
7001 Trondheim
Telefon: (075) 15 660
Telex 55548
Telegram: "NORSHELF"



REPORT TITLE Source rock analyses of well 34/10-5	
CLIENT Statoil	
CLIENT'S REF.: Bjørn Rasmussen	REPORT NO.:

AUTHOR (S): M. Bjorøy, J.O. Vigran	DATE: 28.2.80	PROJECT NO.: 0-247
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DEPARTMENT: Organic Geochemistry	RESPONSIBLE SCIENTIST: Malvin Bjorøy	

SUMMARY:

The analysed sequence, 1790-1910 m was found to be immature with a good potential for gas (and oil). Migrated hydrocarbons are recorded in some of the analysed samples.

KEY WORDS

Source rock

EXPERIMENTAL

One ml. of the headspace gas from each of the cans was analysed gas chromatographically for light hydrocarbons. The results are shown in Table Ia. The canned samples were washed with tempered water on a 0.125 mm sieve to remove drilling mud and thereafter dried at 35⁰C.

Total Organic Carbon (TOC)

The various selected samples were crushed on a centrifugal mill and sieved. The portions with a particle size between 0.125 mm and 0.063 mm were used in the further work. Aliquotes of the samples were treated with hot 6N HCl to remove carbonate and washed twice with distilled water to remove traces of HCl, then placed in a vacuum oven at 50⁰C, evacuated to 20 mm Hg for 12 hrs. The samples were then analysed on a Leco E C 12 carbon determinator, to determine the total organic carbon (TOC).

Extractable Organic Matter (EOM)

From the TOC results samples were selected for extraction. Of the selected samples, approximately 100 gm of each was extracted on soxhlet apparatus for 48 hrs using dichloromethane (DCM) as solvent. The DCM used as solvent was distilled in an all glass apparatus to remove contaminants. The paper thimbles used in the soxhlet apparatus were previously washed with DCM on a large soxhlet apparatus for 48 hrs. to remove any soluble components.

Activated copper foil was used in the flasks to remove any free sulphur from the samples.

After extraction, the solvent was removed on a Buchi Rotavapor and transferred to a 50 ml flask. The rest of the solvent was then removed and the amount of extractable organic matter (EOM) determined.

Chromatographic Separation

The extractable organic matter (EOM) was separated on chromatographic columns, packed with silica, Riedel & Hähn, 0.063 mm, using the slurry method with hexane as solvent. On top of the silica, small amounts of

alumina, approximately 2 cm, was added. The EOM, after it was "taken up" on alumina, was transferred to the top of the columns, which were then eluted with predistilled hexane, benzene and methanol using a ratio of 200 ml of each solvent pr. gm of EOM.

The various eluants were removed on a Buchi Rotavapor and the samples transferred to vials and dried at 40⁰C in a stream of dry nitrogen, and the amount of the various fractions, saturated, aromatic and NSO fraction (Nitrogen, Sulphur, Oxygen), determined. The saturated fractions were analysed gas chromatographically on a 25 m OV 101 glass capillary column with He as carrier gas (0.7 ml/min.) using the splitless injection technique. The glass capillary column was mounted in a Carlo Erba F V 2150 gas chromatograph.

Vitrinite Reflectance

Samples, taken at various intervals, were sent for vitrinite reflectance measurements at Geoconsultants, Newcastle-upon-Tyne. The samples were mounted in Bakelite resin blocks; care being taken during the setting of the plastic to avoid temperatures in excess of 100⁰C. The samples were then ground, initially on a diamond lap followed by two grades of corundum paper. All grinding and subsequent polishing stages in the preparation were carried out using isopropyl alcohol as lubricant, since water leads to the swelling and disintegration of the clay fraction of the samples.

Polishing of the samples was performed on Selvyt cloths using three grades of alumina, 5/20, 3/50 and Gamma, followed by careful cleaning of the surface.

Reflectance determinations were carried out on a Leitz M.P.V. microphotometer under oil immersion, R.I. 1.516 at a wavelength of 546 nm. The field measured was varied to suit the size of the organic particle, but was usually of the order of 2 micron diameter.

The surface of the polished block was searched by the operator for suitable areas of vitrinitic material in the sediment. The reflectance of the organic particle was determined relative to optical glass standards of known reflectance. Where possible, a minimum of twenty individual particles of vitrinite was measured, although in many cases this number could not be achieved.

Processing of Samples for Evaluation of Visual Kerogen

The rock samples were crushed and afterwards treated with hydrochloric and hydrofluoric acids to remove the minerals. A series of microscopic slides was mounted in glycerine jelly:

T-slide represents the total acid insoluble residue.

0-slide represents the residue screened through 15 sieves.

N-1,2,3 slides contain palynodebris remaining after flotation ($Zn Br_2$) to remove disturbing heavy minerals.

X-1,2,3 slides contain oxidized residues, when oxidizing is required due to high coalification or much sapropel.

T & 0 slides are necessary to evaluate kerogen composition/palynofacies which is closely related to sample lithology.

Screened slides are normally required to concentrate the larger fragments, and to study palynomorphs (pollen, spores and dinoflagellates) for paleo-dating and colour evaluation.

So far visual evaluations of kerogen have been undertaken from residues mounted in glycerine jelly, and studied by Leitz Dialux in normal light (halogene) using x10 and x40 objectives.

Rock-Eval Pyrolyses

100 mg crushed sample was put into a boat whose bottom and cover are made of sintered steel and analysed on a Rock-Eval pyrolyser.

Results and discussion

An interval of 120 m (1790 - 1910 m) was analysed. The light hydrocarbon examination shows the samples to have a good abundance of $C_1 - C_4$ hydrocarbons, especially in the lower part. This indicates that hydrocarbons are reservoired in the sandstone in this sequence. The gas is very dry, but the wetness increases in the lower most sample. The isobutane/n-butane ($ic_4/n-C_4$) ratio is high.

The total organic carbon results vary considerably for the claystone (0.79-6.69%). This variation is probably due to coaly particles in the cuttings. On the whole the samples analysed are found to have a good abundance of organic carbon.

Three samples were extracted and analysed chromatographically. (Table III - VII). The uppermost sample, 1805-20 m has a good abundance while the two other samples have a rich abundance of extractable hydrocarbons. The ratio of hydrocarbons to total organic carbon (HC/TOC) is, however, high for these two samples indicating that they might be contaminated with migrated hydrocarbons.

The gas chromatograms of the saturated hydrocarbon fraction vary only slightly.

There are two main differences in the three samples. $n-C_{14}$ is very abundant in the two lowermost samples and they are more naphthenic than the sample from 1805-20 m. The latter indicate that these hydrocarbons come from an amorphous source.

Three samples were analysed for vitrinite reflectance. In the following these three samples are described and other information from the analyses is given.

1790-1805: Calcareous shale and limestone. $R_o = 0.47$ (3) and $R_o = 0.70$ (2).

The sample contained only a few cuttings of true sediments showing particles, mostly of inertinite and reworked material. Only a few particles of possibly true vitrinite. UV light shows a yellow/orange fluorescence from spores and a trace of exinite.

1835-50 m: Shale and carbonate, $R_o = 0.44$ (20).

The sample has a low to moderate organic content. Particles of vitrinite and inertinite but mostly reworked material. UV light shows a yellow and yellow to orange fluorescence from spores and hydrocarbon specs together with a trace of exinite.

1880-95 m. Limestone, and shale, $R_o = 0.48$ (9), $R_o = 0.66$ (7) and $R_o = 1.17$ (10).

The sample, which contains large pyrite masses, has a moderate overall organic content with loose coal fragments of extremely variable reflectance. Some particles in shale of lower reflectance and in limestone of higher reflectance. UV light shows a yellow to orange fluorescence from spores in a few cuttings and a low overall exinite content. With the background in the fluorescence measurements, the lower reflectance value is believed to be the true value.

Material from five samples, 1790-1805 m, 1805-1820 m, 1835-1850 m, 1865-1880 m and 1180-1895 m were selected for analysis of the acid insoluble organic remains.

The material was not well suited for kerogen analysis since the samples gave only sparse residues.

1790-1805 m and 1805-1820 m: The residue apparently contain only artifacts; walnut shells and chemical mud additives.

Colour index: Not determined.

1835-1850 m: The residue contains chemical mud additives. Small organic fragments are partly embedded in the mud additives.

Tertiary fossils are recorded in this sample. These probably come from cavings. Indeterminate herbaceous material and some woody matter may belong to these cuttings.

Colour index: Tentatively put as 2/2+. The value may be based on reworked material and thus too high as a maturation index.

1865-1880 m and 1880-1895 m: The residues clearly differ from those above; they are finely dispersed, apparently consisting of equal parts of true sapropel and herbaceous material. The distinction is difficult due to small particles. After screening, cysts, cuticles, pollen, spores and indeterminate material remain in the residue, together with mud additives.

Colour index: 2.

Remark: We do not feel confident that the exines used for colour evaluation belong to this interval.

Five samples were analysed on a Rock-Eval instrument (Table IX). The samples are found to be immature. The upper four samples have moderate hydrogen indices and high oxygen indices, which indicate that they are in the coal zone, i.e. they respond during pyrolyses as coal would do. Probably due to the samples containing coaly material. The three lowermost samples have low hydrogen and high oxygen indices, typical for immature type III kerogen. The samples from 1820-1880 m have a large production indices, indicating migrated hydrocarbons.

CONCLUSION

The claystone in the analysed sequence is found to be immature; with a good potential for gas (and oil). Migrated hydrocarbons are recorded in some of the analysed samples.

TABLE I

Sample	Depth (m)	C ₁	C ₂	C ₃	iC ₄	nC ₄	C ₅ ⁺	ΣC ₁ -C ₄	ΣC ₂ -C ₄	% wetness	iC ₄ / nC ₄
K 1652	1790 - 1805	57506	4097	1269	626	403	139	62903	6296	10.01	1.55
K 1653	1805 - 20	40847	2926	1064	695	459	663	45992	5145	11.19	1.52
K 1654	1820 - 35	24362	1810	737	541	362	1069	27812	3450	12.41	1.49
K 1655	1835 - 50	7010	362	118	105	72	1389	7667	657	8.56	1.46
K 1656	1850 - 65	123362	15080	4544	3212	2140	3214	148340	24977	16.84	1.50
K 1657	1865 - 80	195297	25255	5574	3529	2343	4309	231997	36700	15.82	1.51
K 1658	1880 - 95	121379	13841	2321	1417	1026	1717	139984	18604	13.29	1.38
K 1659	1895 - 1910	26309	8649	2438	1707	1357	2385	40460	14151	34.98	1.26

tabel 1

T A B L E II

IKU No.	Depth	TOC	Lithology
k 1652	1790 - 1805	2.23	40% Claystone, light grey; 60% Limestone, white; sm.am Mica; Sandstone, light brownish, coarse - fine; Pyrite
K 1653	1805 - 20	6.69	50% Claystone, light grey, brownish; 50% Limestone, white-light brown; sm.am Pyrite; Coal; Sandstone, some calcarious white-brown, coarse-fine, Mica;
K 1654	1820 - 35	2.09	70% Claystone, light grey; 30% Limestone, white; sm.am Coal; Pyrite; Mica;
K 1655	1835 - 50	0.94	60% Claystone, light grey, green; 30% Limestone, white-grey; 20% Sand, white subrounded fine-coarse; sm.am Pyrite; Coal; Mica;
K 1656	1850 - 65	0.79	20% Claystone, light grey, brown; 40% Limestone, white; 30% Pyrite; 10% Sand, white, rounded, coarse; sm.am Glauconite; Mica; Coal;
K 1657	1865 - 80	4.51	60% Claystone, light grey, brown; 25% Calc/Sandstone, white-light brown; 10% Limestone, white; 5% Pyrite; sm.am Mica; Glauconite;
K 1658	1880 - 95	2.60	30% Claystone, light grey, brown; 40% Sand, white, subrounded-rounded, fine-coarse; 10% Limestone, white; 20% Pyrite; sm.am Glauconite; Coal;

T A B L E II

IKU No.	Depth	TOC	Lithology
K 1659	1895 - 1910		2% Claystone, light grey; 4% Pyrite; 94% Sand, white, subrounded-rounded, coarse-fine; sm.am. Coal;

TABLE III

WEIGHT (mg) OF EOM AND CHROMATOGRAPHIC FRACTIONS

IKU No.	Depth	Rock extracted (g)	EOM	Sat.	Aro.	HC	Non HC	TOC
K 1653	1805-20	38.0	44.1	7.5	14.8	22.3	10.1	6.69
K 1654	1820-35	26.1	145.9	11.6	48.9	60.5	46.8	2.09
K 1656	1850-65	28.4	52.9	6.6	17.6	24.2	13.5	0.79

TABLE IV

CONCENTRATION OF EOM AND CHROMATOGRAPHIC FRACTIONS (Weight ppm of rock)

IKU No.	Depth	EOM	Sat.	Aro.	HC	Non HC
K 1653	1805-20	1160	197	389	587	266
K 1654	1820-35	5590	444	1874	2318	1793
K 1656	1850-65	1863	232	620	852	475

TABLE V

CONCENTRATION OF EOM AND CHROMATOGRAPHIC FRACTIONS (mg/g/TOC).

IKU No.	Depth (m)	EOM	SAT	Aro	Total hydrocarb.	Non hydrocarb.
K 1653	1805-20	17.4	3.0	5.8	8.8	4.0
K 1654	1820-35	267.5	21.3	89.6	110.9	85.8
K 1656	1850-65	235.8	29.4	78.5	107.9	60.2

TABLE VI

COMPOSITION IN % OF THE MATERIAL EXTRACTED FROM THE ROCK

IKU No.	Depth (m)	$\frac{\text{Sat}}{\text{EOM}}$	$\frac{\text{Aro}}{\text{EOM}}$	$\frac{\text{HC}}{\text{EOM}}$	$\frac{\text{Sat}}{\text{Aro}}$	$\frac{\text{Non HC}}{\text{EOM}}$	$\frac{\text{HC}}{\text{Non HC}}$
K 1653	1805-20	17	34	51	51	23	221
K 1654	1820-35	8	34	41	24	32	129
K 1656	1850-65	12	33	46	38	26	179

TABLE VII

TABULATION OF DATAS FROM THE GASCHROMATOGRAMS

IKU No.	Depth (m)	Pristane/nC ₁₇	Pristane/Phytane	CPI
K 1653	1805-20	0.46	1.22	1.0
K 1654	1820-35	0.49	1.17	1.0
K 1656	1850-65	0.64	1.29	1.0

TABLE VIII

VITRINITE REFLECTANCE AND VISUAL KEROGEN MEASUREMENTS

IKU No.	Depth	Vitrinite Reflectance	Colour Index	Type of Organic Matter
K 1652	1790-1805	0.47(3), 0.70(2)		Barren
K 1653	1805-20			Barren
K 1655	1835-50	0.44(20)	2/2+ ?	Almost barren
K 1656	1865-80		2	Am/He
K 1658	1880-95	0.48(9), 0.66(7), 1.17(10)	2	Am/He

TABLE IX

ROCK-EVAL PYROLYSES

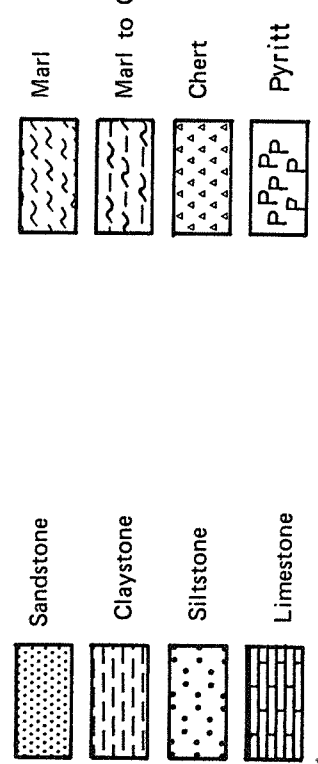
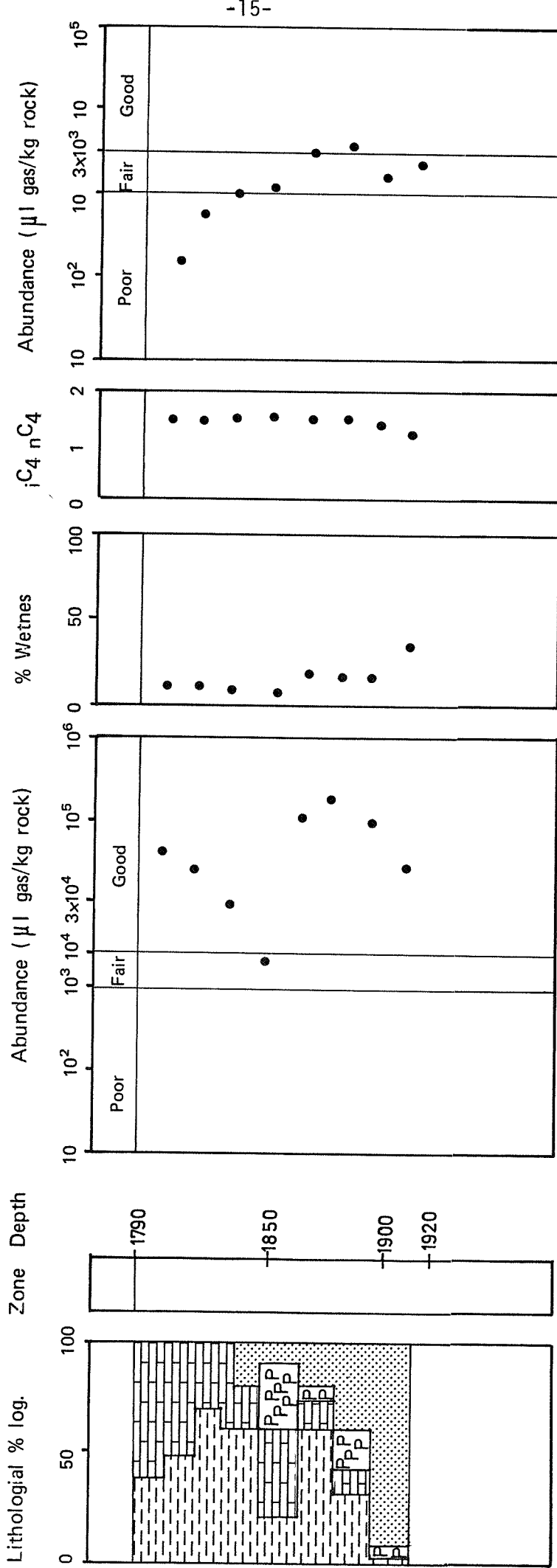
Sample	Depth	S ₁	S ₂	S ₃	C _{org}	Hydrogen Index	Oxygen Index	Oil of gas content (S ₁ + S ₂)	Production Index $\frac{S_1}{S_1 + S_2}$	T _{max} °C
K 1652	1790-1805	0.49	2.32	4.15	2.23	104.04	186.10	2.81	0.17	419
K 1653	1805-20	2.69	16.54	13.58	6.69	247.24	202.99	19.23	0.14	428
K 1654	1820-35	1.41	2.04	2.81	2.09	97.61	134.45	3.45	0.41	419
K 1655	1835-50	0.62	1.37	1.84	0.94	145.75	195.75	1.99	0.31	434
K 1656	1850-65	0.19	0.12	1.78	0.79	15.19	225.32	0.31	0.61	432
K 1657	1865-80	4.95	2.20	7.64	4.51	28.80	169.40	7.15	0.69	427
K 1658	1880-95	0.11	1.18	4.53	2.60	45.38	174.23	1.29	0.09	427

C₁ - C₇ HYDROCARBONS

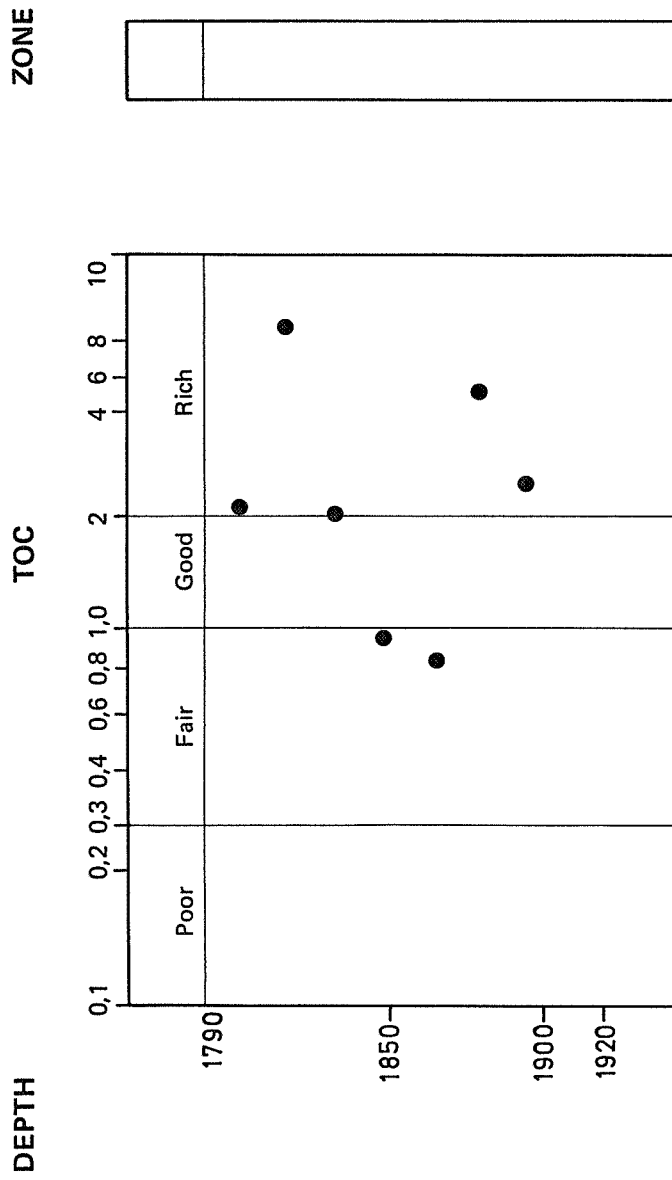
Presentation of Analytical Data

C₁ - C₄ HYDROCARBONS

C₅ - C₇ HYDROCARBONS

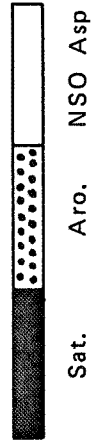
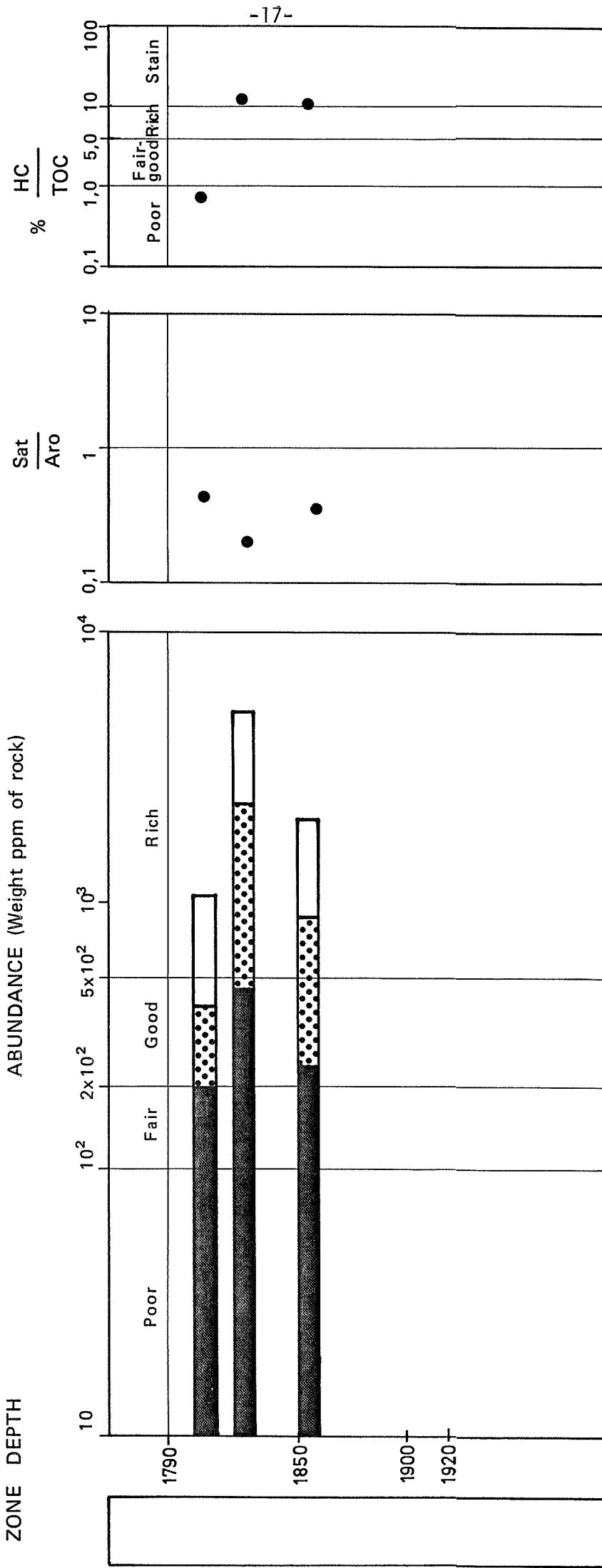


TOTAL ORGANIC CARBON (TOC)
Presentation of Analytical Data



C₁₅⁺ HYDROCARBONS

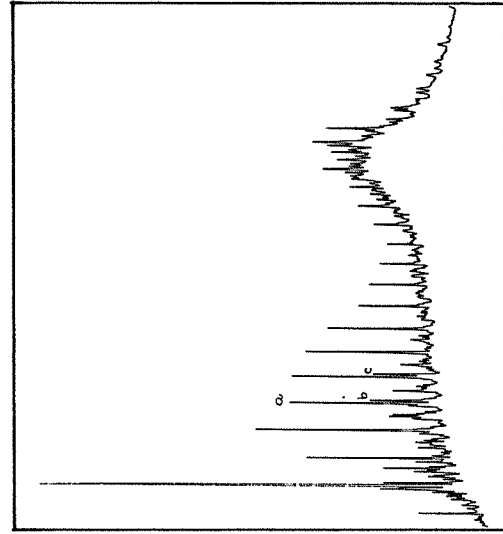
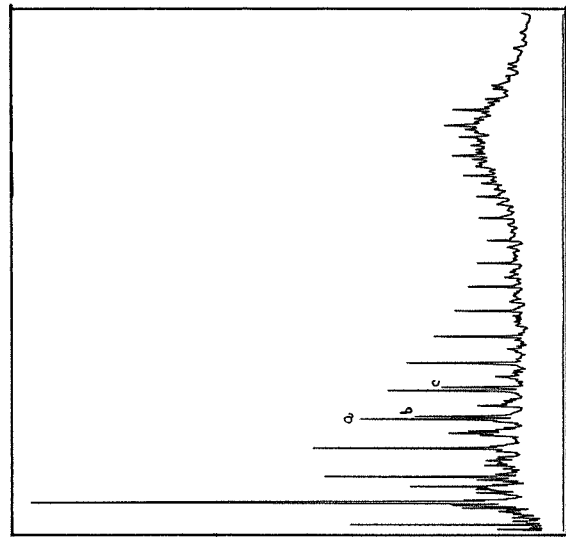
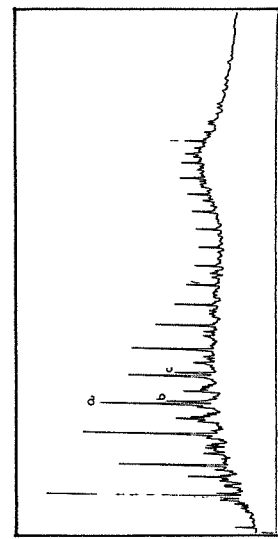
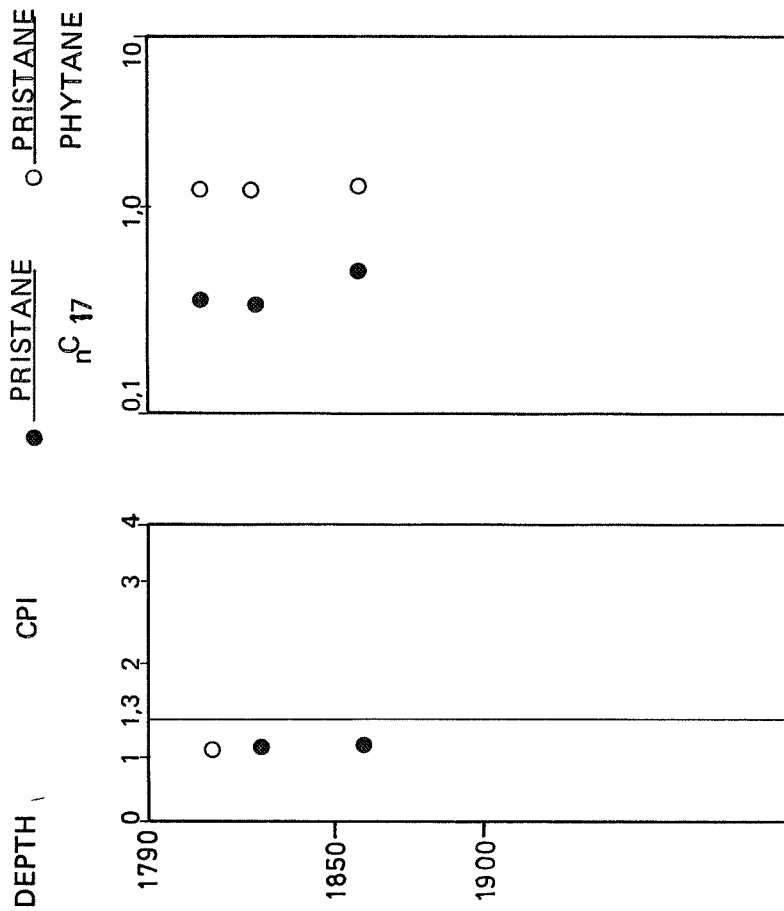
Presentation of Analytical Data



Sat: Saturated Hydrocarbons
 Aro: Aromatic Hydrocarbons
 NSO: Nitrogen, Sulphur and Oxygen containing compounds

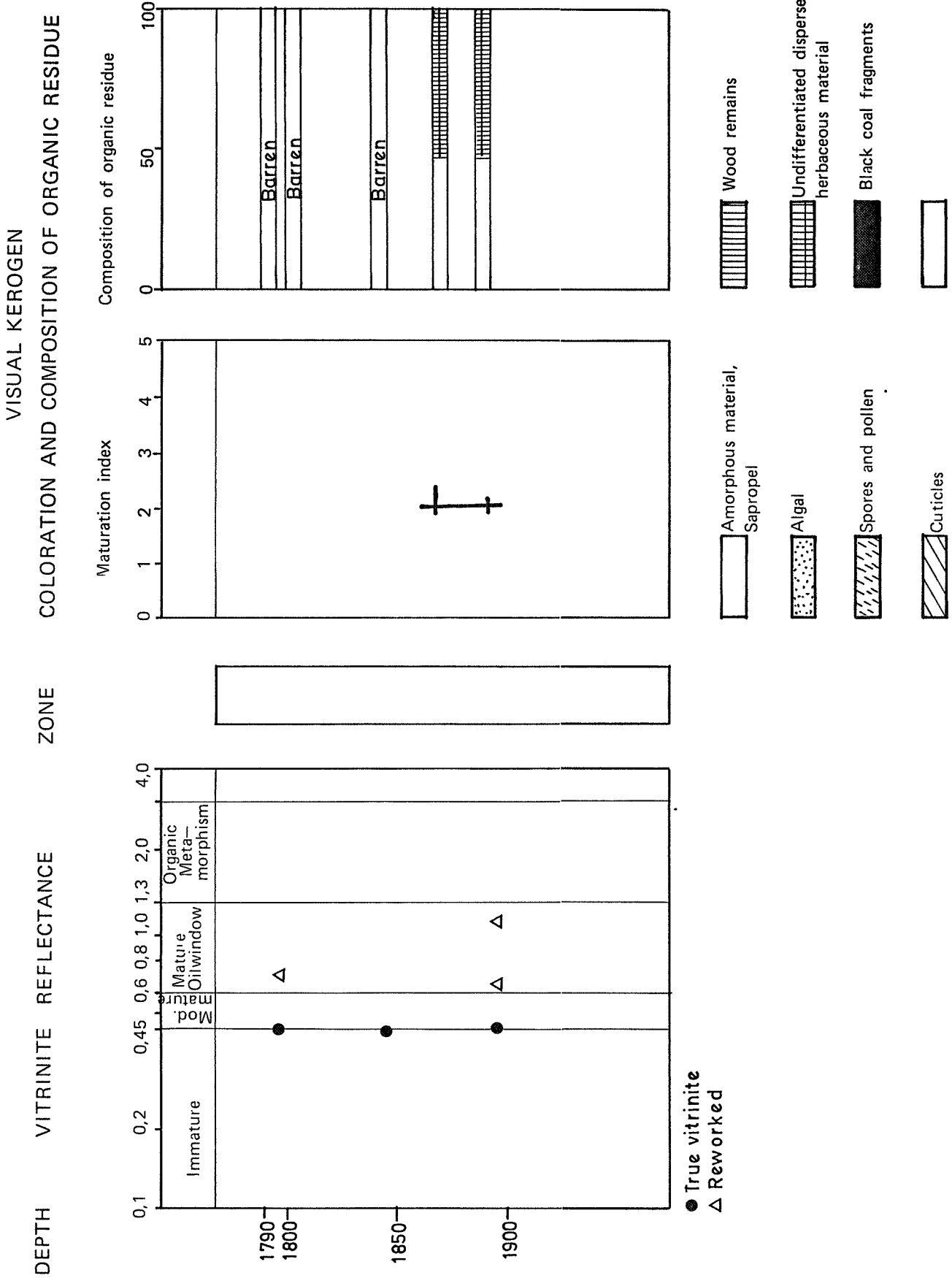
Asp: Asphaltenes
 HC: C₁₅ Hydrocarbons
 TOC: Total Organic Carbon

C₁₅⁺ SATURATED HYDROCARBONS



a = nC₁₇
 b = Pristane
 c = Phytane

MATURATION



DEPTH

VITRINITE REFLECTANCE

ZONE

COLORATION AND COMPOSITION OF ORGANIC RESIDUE

Composition of organic residue

Maturation index

1790
1800
1850
1900

0
50
100

0
1
2
3
4
5

0,1
0,2
0,45
0,6
0,8
1,0
1,3
2,0
4,0

Immature
Mod. mature
Mature Oil window
Organic Meta-morphism

Barren
Barren
Barren

● True vitrinite
△ Reworked

Wood remains

Amorphous material, Sapropel

Algal

Undifferentiated disperse herbaceous material

Spores and pollen

Black coal fragments

Cuticles